

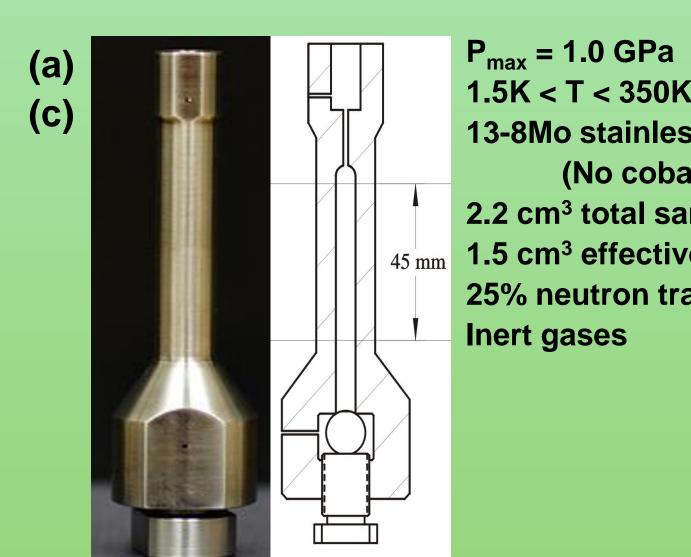


Hydrostatic High Pressure Capabilities at the NIST Center for Neutron Research

Juscelino B. Leão

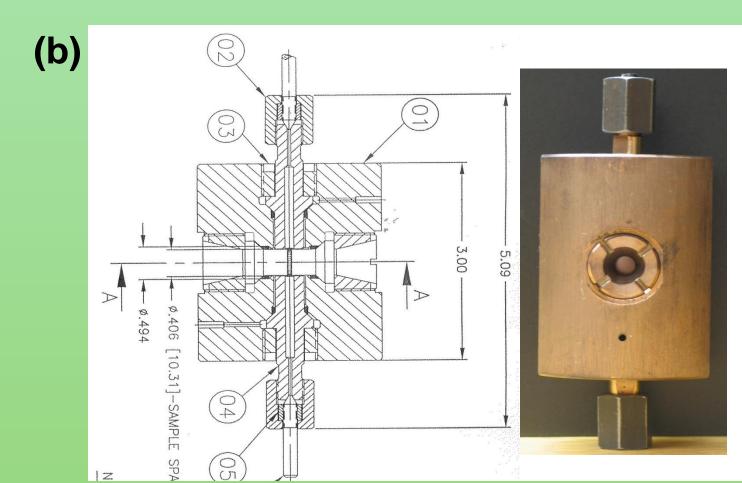
NIST Center for Neutron Research. Gaithersburg, MD

The NIST Center for Neutron Research currently provides a series of pressure apparatus ranging to as high as 2.5 GPa that are specially designed for neutron spectroscopy. Most of the pressure equipment can be mounted in a variety of instruments throughout NCNR's facility, allowing for experimental flexibility and maximizing beam time use.



1.0 GPa Inert Gas Pressure Vessel

13-8Mo stainless steel construction (No cobalt) 2.2 cm³ total sample volume 1.5 cm³ effective illuminated volume 25% neutron transmission at 2Å



300 MPa Sapphire Window Pressure Vessel

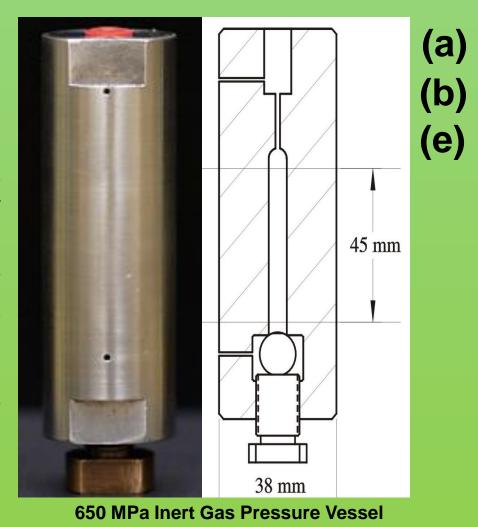
 $P_{max} = 300 MPa$

 $LN_2 < T < 350K$

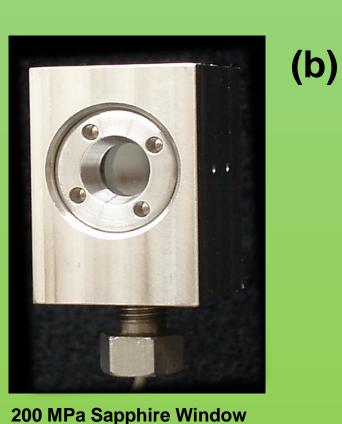
CuBe construction with sapphire windows 2.2 cm³ total sample volume Neutron beam cross-section area 0.3 cm²

Beam divergence angle $\theta \approx 15^{\circ}$ **Inert gases**

 $P_{\text{max}} = 650 \text{ MPa}$ 1.5K < T < 300K **Aluminum 7075-T6 construction** 1.5 cm³ sample volume .635 cm dia. x 5.08 cm sample illumination 60% Neutron Transmission at 2Å **Inert Gases**



 $P_{\text{max}} = 200 \text{ MPa}$ $LN_2 < T < 350K$ S.S. Construction with sapphire windows 1.0 Cm² total sample cross section area Adjustable sample thickness Beam divergence angle $\theta \approx 20^{\circ}$ Methane, CO₂, H₂, inert gases, fluids



SANS Pressure Vessel

(d)

Air sensitive/gas loading $P_{max} = 1.4 MPa (V)$ **Up to 0.50 MPa (al)** $1.5k \le T \le 800k$ Heated gas line available for methane and CO₂

Air sensitive closure with vanadium sample can

 $P_{\text{max}} = 2.5 \text{ GPa}$ $1.5K \le T \le 300K$ Sample Size: 10 mm x 6 mm Ø 140 mm (a)

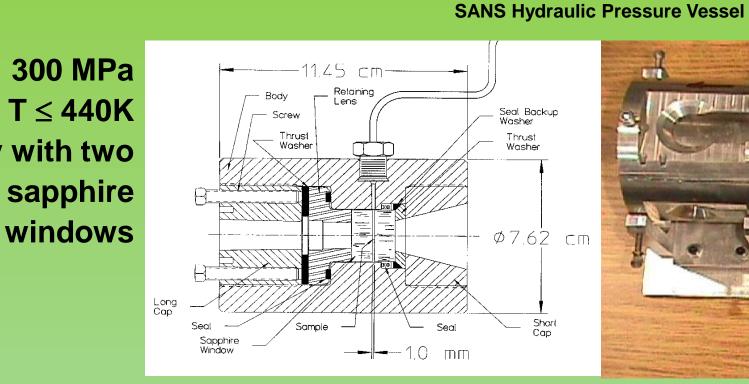
McWhan Clamp Cell schematics and as seen mounted on SD-55 (below)

The sample is axially pressurized between two opposing cylindrical **Tungsten Carbide pistons.** Hydrostatic uniformity is ensured

by immersing the sample in a pressure transmitting media such as Fluorinert (C_8F_{18}), or a 4:1 mixture of deuterated methanol and ethanol

Pressure is monitored through the change in lattice parameters of **NaCl**

 $P_{\text{max}} = 300 \text{ MPa}$ $258K \le T \le 440K$ S.S. body with two containment sapphire





This vessel can be used to pressurize polymer melt mixtures in a wafer form (blends and copolymers confined by an encapsulated o-ring) or soft macromolecular fluids (solutions, micellar systems).

Also used to pressurize flowing liquids though the use of a separator (high pressure tubing containing a piston between the pressurizing fluid and the sample).

Partial list of Citations:

- a) T. Hong, et al. "Neutron scattering study of a quasi-2D spin-1/2 dimer system Piperazinium Hexachlorodicuprate under hydrostatic pressure." IN PRESS.
- b) C. Tsao, et al. "Neutron Scattering Methodology for Absolute Measurement of Room-Temperature Hydrogen Storage Capacity and Evidence for Spillover Effect in a Pt-Doped Activated Carbon." J. Phys. Chem. Lett. 2010, 1, 1569–1573.
- c) N. P. Butch, et al. "Antiferromagnetic critical pressure in URu2Si2 under hydrostatic conditions." Phys. Rev. B 82, 060408 R 2010.
- C.M. Brown, et al. "Hydrogen adsorption in HKUST-1: a combined inelastic neutron scattering and first-principles study", Nanotechnology 20, 204025 (2009).
- A. Kreyssig, et al. "Pressure-induced volume-collapsed tetragonal phase of CaFe2As2 as seen via neutron scattering." Physical Review B 78, 184517 (2008)
- A.J. Patel, et al. "Observing Nucleation Close to the Binodal by Perturbing Metastable Polymer Blends." *Macromol.* **40**(5), 1675 (2007).